

dis his

(FILE 'HOME' ENTERED AT 10:06:22 ON 07 SEP 2007)

FILE 'REGISTRY' ENTERED AT 10:06:38 ON 07 SEP 2007  
E RN=119

FILE 'REGISTRY' ENTERED AT 10:16:31 ON 07 SEP 2007

L1 1 S 119-58-4/RN  
L2 8823 S AMMONIA?

FILE 'CAPLUS' ENTERED AT 10:17:06 ON 07 SEP 2007

L3 39100 S S1  
L4 36445 S S2  
L5 281 S L1  
L6 282422 S L2  
L7 0 S L5 (5P) L6  
L8 4 S L5 AND L6

FILE 'REGISTRY' ENTERED AT 10:20:43 ON 07 SEP 2007

L9 1 S 119-58-4?/RN  
L10 0 S (AMINE OR POLYAMINE OR AMMONIA? OR DIAMINE)/RN  
L11 0 S AMMONIA?/RN OR AMINE/RN OR POLYAMINE/RN OR DIAMINE/RN  
L12 0 S AMMONIA?/RN  
L13 8823 S AMMONIA?

FILE 'CAPLUS' ENTERED AT 10:23:08 ON 07 SEP 2007

L14 281 S L9  
L15 282422 S L13  
L16 4 S L14 AND L15

FILE 'REGISTRY' ENTERED AT 10:28:27 ON 07 SEP 2007

L17 8 S MICHLER?/CN  
L18 1687 S AMMONIA?/CN

FILE 'CAPLUS' ENTERED AT 10:29:06 ON 07 SEP 2007

L19 4208 S L17  
L20 262585 S L18  
L21 27 S L19 AND L20 NOT L8

=> s l21 and (breath? or gas? or vapor? or air? or exhal?)

38315 BREATH?  
2144354 GAS?  
626839 VAPOR?  
1087274 AIR?  
7144 EXHAL?

L22 2 L21 AND (BREATH? OR GAS? OR VAPOR? OR AIR? OR EXHAL?)

=> dis bib hit kwic 1-2 L22

L22 ANSWER 1 OF 2 CAPLUS COPYRIGHT 2007 ACS on STN

AN 1986:79230 CAPLUS <<LOGINID::20070907>>

DN 104:79230

TI Image formation

IN Irving, Edward; Smith, Terence James

PA Ciba-Geigy A.-G. , Switz.

SO Eur. Pat. Appl., 36 pp.

CODEN: EPXXDW

DT Patent

LA German

FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	EP 146505	A2	19850626	EP 1984-810618	19841214

EP 146505	A3	19870603		
EP 146505	B1	19900404		
R: BE, CH, DE, FR, GB, IT, LI, NL, SE				
US 4634644	A	19870106	US 1984-679973	19841210
CA 1246384	A1	19881213	CA 1984-470388	19841218
ES 538836	A1	19861016	ES 1984-538836	19841219
JP 60158441	A	19850819	JP 1984-269594	19841220
PRAI GB 1983-33853	A	19831220		

- AB A process for image formation by means of vapor permeation followed by irradiation with actinic light involves coating a support with a liquid composition that upon contact with a gaseous polymerization agent is hardened, but remains photocrosslinkable, exposing the hardened layer to actinic light through a pattern to achieve crosslinking in the exposed areas, and then developing by removing the nonphotocrosslinked areas with a suitable solvent. Thus, a mixture containing 2,2-bis(4-glycidyoxyphenyl)propane 1, Et 2-cyanoacrylate 1, diphenyliodonium hexafluorophosphate 0.2, and 2-isopropylthioxanthone 0.05 part was coated on a Cu-clad laminate at 10  $\mu$ m thickness. The coating was treated with gaseous NH<sub>3</sub> for 10 s to give a nontacky film and then exposed 40 s at 75 cm through a neg. with a 5000 W metal halide lamp. After development in a mixture of xylene and Me<sub>2</sub>CO (9:1 volume ratio), a good image was obtained.
- IT Phenolic resins, uses and miscellaneous  
 RL: USES (Uses)  
 (glycidyl ethers, esters, photocrosslinkable photoimaging compns. hardenable by vapor permeation containing, for printed elec. circuit fabrication)
- IT Amines, uses and miscellaneous  
 RL: USES (Uses)  
 (in hardening of photocrosslinkable photoimaging compns. by vapor permeation)
- IT Printing plates  
 (photocrosslinkable compns. hardenable by vapor permeation in fabrication of)
- IT Epoxy resins, uses and miscellaneous  
 RL: USES (Uses)  
 (photocrosslinkable photoimaging compns. hardenable by vapor permeation containing, for printed elec. circuit fabrication)
- IT Epoxides  
 RL: USES (Uses)  
 (photocrosslinkable photoimaging compns. hardenable by vapor phase permeation containing)
- IT Amines, uses and miscellaneous  
 Sulfides, uses and miscellaneous  
 RL: USES (Uses)  
 (cyclic, photocrosslinkable photoimaging compns. hardenable by vapor phase permeation containing)
- IT Photoimaging compositions and processes  
 (photocrosslinkable, hardenable by vapor permeation)
- IT Electric circuits  
 (printed, photocrosslinkable compns. hardenable by vapor permeation in fabrication of)
- IT 75-91-2 80-15-9 58495-52-6  
 RL: USES (Uses)  
 (in hardening of photocrosslinkable photoimaging composition by vapor phase permeation)
- IT 108-01-0 7446-09-5, uses and miscellaneous 7637-07-2, properties 7647-01-0, properties 7664-41-7, uses and miscellaneous 7722-84-1, properties 7732-18-5, vapor  
 RL: USES (Uses)  
 (in hardening of photocrosslinkable photoimaging compns. by vapor permeation)
- IT 102-71-6, properties 121-44-8, properties  
 RL: PRP (Properties)

(in hardening of photocrosslinkable photoimaging compns. by vapor permeation)

IT 68-11-1D, esters 79-10-7, uses and miscellaneous 79-41-4, uses and miscellaneous 79-42-5D, esters 96-05-9 107-96-0D, esters 999-55-3 15625-89-5 25119-62-4  
 RL: USES (Uses)  
 (photocrosslinkable photoimaging compns. hardenable by vapor permeation containing)

IT 80-05-7, uses and miscellaneous 90-94-8 101-68-8 104-15-4, uses and miscellaneous 106-91-2D, reaction products with formaldehyde-phenol copolymers 108-55-4D, reaction products with butanediol diglycidyl ether and di(hydroxyphenyl)pentadienone 137-05-3 584-84-9 621-82-9D, esters with formaldehyde-phenol copolymer glycidyl ethers 923-26-2D, ethers with formaldehyde-phenol copolymers 1675-54-3 1745-89-7 2386-87-0 2425-79-8 2425-79-8D, reaction products with di(hydroxyphenyl)pentadienone and glutaric anhydride 3654-49-7D, reaction products with butanediol diglycidyl ether and glutaric anhydride 5495-84-1 7085-85-0 9003-35-4D, glycidyl ethers, cinnamates 9050-83-3 10193-99-4 15625-89-5 24650-42-8 28679-16-5 58109-40-3 100235-51-6  
 RL: USES (Uses)  
 (photocrosslinkable photoimaging compns. hardenable by vapor permeation containing, for printed elec. circuit fabrication)

IT 107-21-1D, esters with thioacids 7440-21-3D, organic compds. 9011-05-6 25852-49-7 52496-08-9  
 RL: USES (Uses)  
 (photocrosslinkable photoimaging compns. hardenable by vapor phase permeation containing)

AB A process for image formation by means of vapor permeation followed by irradiation with actinic light involves coating a support with a liquid composition that upon contact with a gaseous polymerization agent is hardened, but remains photocrosslinkable, exposing the hardened layer to actinic light through a pattern to achieve crosslinking in the exposed areas, and then developing by removing the nonpholocrosslinked areas with a suitable solvent. Thus, a mixture containing 2,2-bis(4-glycidyloxyphenyl)propane 1, Et 2-cyanoacrylate 1, diphenyliodonium hexafluorophosphate 0.2, and 2-isopropylthioxanthone 0.05 part was coated on a Cu-clad laminate at 10  $\mu$ m thickness. The coating was treated with gaseous NH<sub>3</sub> for 10 s to give a nontacky film and then exposed 40 s at 75 cm through a neg. with a 5000 W metal halide lamp. After development in a mixture of xylene and Me<sub>2</sub>CO (9:1 volume ratio), a good image was obtained.

IT Phenolic resins, uses and miscellaneous  
 RL: USES (Uses)  
 (glycidyl ethers, esters, photocrosslinkable photoimaging compns. hardenable by vapor permeation containing, for printed elec. circuit fabrication)

IT Amines, uses and miscellaneous  
 RL: USES (Uses)  
 (in hardening of photocrosslinkable photoimaging compns. by vapor permeation)

IT Printing plates  
 (photocrosslinkable compns. hardenable by vapor permeation in fabrication of)

IT Epoxy resins, uses and miscellaneous  
 RL: USES (Uses)  
 (photocrosslinkable photoimaging compns. hardenable by vapor permeation containing, for printed elec. circuit fabrication)

IT Epoxides  
 RL: USES (Uses)  
 (photocrosslinkable photoimaging compns. hardenable by vapor phase permeation containing)

IT Amines, uses and miscellaneous  
 Sulfides, uses and miscellaneous

RL: USES (Uses)  
(cyclic, photocrosslinkable photoimaging compns. hardenable by  
vapor phase permeation containing)

IT Photoimaging compositions and processes  
(photo-crosslinkable, hardenable by vapor permeation)

IT Electric circuits  
(printed, photocrosslinkable compns. hardenable by vapor  
permeation in fabrication of)

IT 75-91-2 80-15-9 58495-52-6  
RL: USES (Uses)  
(in hardening of photocrosslinkable photoimaging composition by  
vapor phase permeation)

IT 108-01-0 7446-09-5, uses and miscellaneous 7637-07-2, properties  
7647-01-0, properties 7664-41-7, uses and miscellaneous  
7722-84-1, properties 7732-18-5, vapor  
RL: USES (Uses)  
(in hardening of photocrosslinkable photoimaging compns. by  
vapor permeation)

IT 102-71-6, properties 121-44-8, properties  
RL: PRP (Properties)  
(in hardening of photocrosslinkable photoimaging compns. by  
vapor permeation)

IT 68-11-1D, esters 79-10-7, uses and miscellaneous 79-41-4, uses and  
miscellaneous 79-42-5D, esters 96-05-9 107-96-0D, esters 999-55-3  
15625-89-5 25119-62-4  
RL: USES (Uses)  
(photo-crosslinkable photoimaging compns. hardenable by vapor  
permeation containing)

IT 80-05-7, uses and miscellaneous 90-94-8 101-68-8 104-15-4,  
uses and miscellaneous 106-91-2D, reaction products with  
formaldehyde-phenol copolymers 108-55-4D, reaction products with  
butanediol diglycidyl ether and di(hydroxyphenyl)pentadienone 137-05-3  
584-84-9 621-82-9D, esters with formaldehyde-phenol copolymer glycidyl  
ethers 923-26-2D, ethers with formaldehyde-phenol copolymers 1675-54-3  
1745-89-7 2386-87-0 2425-79-8 2425-79-8D, reaction products with  
di(hydroxyphenyl)pentadienone and glutaric anhydride 3654-49-7D,  
reaction products with butanediol diglycidyl ether and glutaric anhydride  
5495-84-1 7085-85-0 9003-35-4D, glycidyl ethers, cinnamates  
9050-83-3 10193-99-4 15625-89-5 24650-42-8 28679-16-5 58109-40-3  
100235-51-6  
RL: USES (Uses)  
(photo-crosslinkable photoimaging compns. hardenable by vapor  
permeation containing, for printed elec. circuit fabrication)

IT 107-21-1D, esters with thioacids 7440-21-3D, organic compds. 9011-05-6  
25852-49-7 52496-08-9  
RL: USES (Uses)  
(photo-crosslinkable photoimaging compns. hardenable by vapor  
phase permeation containing)

L22 ANSWER 2 OF 2 CAPLUS COPYRIGHT 2007 ACS on STN

AN 1949:20718 CAPLUS <<LOGINID::20070907>>

DN 43:20718

OREF 43:3963g-i,3964a-e

TI Antifouling research, 1942-44

AU Harris, John E.

SO Journal of the Iron and Steel Institute, London (1947), Volume Date 1946,  
154(No. 2), 297P-333P

CODEN: JISIAX; ISSN: 0021-1567

DT Journal

LA Unavailable

AB cf. C.A. 37, 5689.1. Chemical studies of anti-fouling and leaching suggest  
that toxicity is produced by a thin layer of poisonous sea water next to  
the paint surface. These studies are judged chiefly by the growth of  
Ulothrix and Fragilaria after 31 days' exposure of the panel. Rate of

loss of Cu of 10  $\gamma$ /sq. cm./day in the laboratory test indicates adequate antifouling properties. Hg lowers the necessary rate loss of Cu. In the leaching test the painted ground-glass slide is immersed in 60 mL. of filtered sea water through which air is bubbled. The apparatus and method are described. An aliquot of the leachate is used for determination of

Cu

by the use of 2 mL. of a 0.1% solution of Na diethyldithiocarbamate. Zn or heavy slime interferes. Another aliquot is used for determination of Hg by

means

of a solution of 20 mg. per l. of diphenylthiocarbazone in  $\text{CHCl}_3$ . Toxicity is defined as the neg.  $\log_{10}$  of the concentration in g. per mL. required for

50%

mortality of the test organism. The toxicity to crustaceans ranges in decreasing order for the following compds. from 9.0 to 4.6; the toxicity to red, brown, and green seaweed (*Lomentaria articulata*, *Laminaria digitata*, *Enteromorpha intestinalis*) is reported for many of these compds.: Hg(SCN)2, HgCrO4, Cu arsenate, Cu(CN)2, Cu salicylate, Cu(SCN)2, Pb(SCN)2, HgNH2Cl, Hg salicylate, 2,4-diisobutylphenol, 4-nitroso-o-cresol, nitroso-m-cresol, amyl-m-cresol, Et bromoacetate, chlorophenarsazine, Co Hg thiocyanate, di(p-dimethylamino) benzophenone,  $\text{PhHgNO}_3$ , tetramethylthiuram di- and monosulfides, nitrosoresorcinol, Pb salicylate, Sb oxalate,  $\text{CdC}_2\text{O}_4$ , pyrethrum, trichlorophenol (Hg salt), 4-nitroso-1-naphthol,  $\text{PhHgOAc}$ , allyl isothiocyanate, pentachlorophenol and its Cu and Hg salts, derris extract, aminoazotoluene, p-chloro-m-xylene, Cu ethylacetoacetate, chloroisothymol, chlorothymol,  $\text{Na}_2\text{HASO}_3$ , trichlorophenol and its Cu salt, Zn phenyldithiocarbamate, thiocyanobenzothiazole, 4-chloro-2,6-dinitrophenol,  $\text{CuC}_2\text{O}_4$ , carbazole, o-cresotic acid, p-dichlorobenzene, nicotine abietate, phenothiazine, tetraethylthiuram disulfide, methylbenzothiazolethione,  $\text{HgCl}_2$ , salicylanilide, 2-benzothiazyl Me sulfide, chlorinated phenols, m-dinitrobenzene, diphenylamine, o-nitroanisole, o-nitrobenzyl cyanide,  $\text{C}_6\text{H}_4(\text{OH})_2$ , 4-amino-1,2'-azonaphthalene, phenoselenazine, nitrosophenol,  $\text{CuSO}_4$ , Me dimethyldithiocarbamate. The poisonous action of Cu and Hg on crustaceans and on many seaweeds is increased when present as salts of organic acids. Three forms of antifouling coatings were used in a series of small-scale panel tests at Millport: a rosin coal-tar-pitch/linseed oil varnish medium, milled with 50% bauxite residue red and 10% of organic poison; same medium with 40% bauxite residue red, 10% organic poison, and 10%  $\text{Cu}_2\text{O}$ ; a cementiferous coating containing about 30% by volume of the organic poison.

The toxicity, naphtha-solubility, and an estimate of the fouling on these coatings

at 6, 12, and 18 wk after immersion are reported for numerous organic compds. including those of As, Zn, Fe, Cu, and Hg, and thiuram derivs., anilides, phenols, and azo compds.; of these the most effective were diphenylarsenious oxide,  $\beta$ -chlorovinyl arsenious oxide, chlorophenarsazine, 10-ethyl-5,10-dihydrophenarsazine, ferric dimethyldithiocarbamate, Et bromoacetate, and derris extract. The leaching requirements are more easily met for cementiferous than for oleoresinous coatings, because the latter require adjustment of both water- and naphtha-solubility

IT

55-68-5, Mercury, phenyl-, nitrate 57-06-7, Isothiocyanic acid, allyl ester 62-38-4, Mercury, phenyl-, acetate 69-72-7, Salicylic acid, compds. with Cu, Pb and Hg salts 83-40-9, 2,3-Cresotic acid 86-74-8, Carbazole 87-17-2, Salicylanilide 88-04-0, 3,5-Xylenol, 4-chloro-88-87-9, Phenol, 4-chloro-2,6-dinitro- 89-68-9, Thymol, 6-chloro-90-94-8, Benzophenone, 4,4'-bis(dimethylamino)- 91-23-6, Anisole, o-nitro- 92-84-2, Phenothiazine 97-74-5, Sulfide, bis(dimethylthiocarbamoyl)- 97-77-8, Disulfide, bis(diethylthiocarbamoyl) 99-65-0, Benzene, m-dinitro- 105-36-2, Acetic acid, bromo-, ethyl ester 106-46-7, Benzene, p-dichloro-122-39-4, Diphenylamine 137-26-8, Disulfide, bis(dimethylthiocarbamoyl) 262-05-5, Phenoselenazine 578-94-9, Phenarsazine, 10-chloro-5,10-dihydro-

592-85-8, Mercury thiocyanate,  $\text{Hg}(\text{SCN})_2$  592-87-0, Lead thiocyanate,  $\text{Pb}(\text{SCN})_2$  605-60-7, 1-Naphthol, 4-nitroso- 610-66-2, Acetonitrile, (o-nitrophenyl)- 814-88-0, Cadmium oxalate,  $\text{CdC}_2\text{O}_4$  1331-84-6, Carvacrol, chloro- 3735-92-0, Carbamic acid, dimethyldithio-, methyl ester 4367-08-2, Copper cyanide,  $\text{Cu}(\text{CN})_2$  6971-38-6, o-Cresol, 4-nitroso- 7057-72-9, Copper oxalate 7487-94-7, Mercury chloride,  $\text{HgCl}_2$  7758-98-7, Copper sulfate 10124-48-8, Mercury, ammonobasic ( $\text{HgNH}_2\text{Cl}$ ) 13444-75-2, Mercury chromate,  $\text{HgCrO}_4$  13466-06-3, Sodium arsenite,  $\text{Na}_2\text{HAsO}_3$  15443-74-0, Cobalt mercury thiocyanate 16455-98-4, Antimony oxalate 25167-82-2, Phenol, trichloro- 25167-82-2, Phenol, trichloro-, mercury derivative 25500-26-9, Carbanilic acid, dithio-, zinc salt 26856-15-5, Phenol, trichloro-, copper salt 29383-39-9, 2-Benzothiazolinethione, methyl- 29871-13-4, Copper arsenate 44612-23-9, Copper thiocyanate,  $\text{Cu}(\text{SCN})_2$  63041-98-5, 1-Naphthylamine, 4-(2-naphthylazo)- 65152-07-0, Phenol, 2,4-diisobutyl- 91588-45-3, m-Cresol, nitroso- 102763-39-3, Phenol, nitroso- 720688-66-4, Nicotine, compound with abietic acid 720688-66-4, Abietic acid, compound with nicotine

(antifouling properties of)

AB cf. C.A. 37, 5689.1. Chemical studies of anti-fouling and leaching suggest that toxicity is produced by a thin layer of poisonous sea water next to the paint surface. These studies are judged chiefly by the growth of *Ulothrix* and *Fragilaria* after 31 days' exposure of the panel. Rate of loss of Cu of 10  $\gamma$ /sq. cm./day in the laboratory test indicates adequate antifouling properties. Hg lowers the necessary rate loss of Cu. In the leaching test the painted ground-glass slide is immersed in 60 mL. of filtered sea water through which air is bubbled. The apparatus and method are described. An aliquot of the leachate is used for determination of

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IT 55-68-5, Mercury, phenyl-, nitrate 57-06-7, Isothiocyanic acid, allyl ester 62-38-4, Mercury, phenyl-, acetate 69-72-7, Salicylic acid, compds. with Cu, Pb and Hg salts 83-40-9, 2,3-Cresotic acid 86-74-8, Carbazole 87-17-2, Salicylanilide 88-04-0, 3,5-Xylenol, 4-chloro- 88-87-9, Phenol, 4-chloro-2,6-dinitro- 89-68-9, Thymol, 6-chloro- 90-94-8, Benzophenone, 4,4'-bis(dimethylamino)- 91-23-6, Anisole, o-nitro- 92-84-2, Phenothiazine 97-74-5, Sulfide, bis(dimethylthiocarbamoyl)- 97-77-8, Disulfide, bis(diethylthiocarbamoyl) 99-65-0, Benzene, m-dinitro- 105-36-2, Acetic acid, bromo-, ethyl ester 106-46-7, Benzene, p-dichloro- 122-39-4, Diphenylamine 137-26-8, Disulfide, bis(dimethylthiocarbamoyl) 262-05-5, Phenoselenazine 578-94-9, Phenarsazine, 10-chloro-5,10-dihydro- 592-85-8, Mercury thiocyanate, Hg(SCN)<sub>2</sub> 592-87-0, Lead thiocyanate, Pb(SCN)<sub>2</sub> 605-60-7, 1-Naphthol, 4-nitroso- 610-66-2, Acetonitrile, (o-nitrophenyl)- 814-88-0, Cadmium oxalate, CdC<sub>2</sub>O<sub>4</sub> 1331-84-6, Carvacrol, chloro- 3735-92-0, Carbamic acid, dimethyldithio-, methyl ester 4367-08-2, Copper cyanide, Cu(CN)<sub>2</sub> 6971-38-6, o-Cresol, 4-nitroso- 7057-72-9, Copper oxalate 7487-94-7, Mercury chloride, HgCl<sub>2</sub> 7758-98-7, Copper sulfate 10124-48-8, Mercury, ammonobasic (HgNH<sub>2</sub>Cl) 13444-75-2, Mercury chromate, HgCrO<sub>4</sub> 13466-06-3, Sodium arsenite, Na<sub>2</sub>HAsO<sub>3</sub> 15443-74-0, Cobalt mercury thiocyanate 16455-98-4, Antimony oxalate 25167-82-2, Phenol, trichloro- 25167-82-2, Phenol, trichloro-, mercury derivative 25500-26-9, Carbanilic acid, dithio-, zinc salt 26856-15-5, Phenol, trichloro-, copper salt 29383-39-9, 2-Benzothiazolinethione, methyl- 29871-13-4, Copper arsenate 44612-23-9, Copper thiocyanate, Cu(SCN)<sub>2</sub> 63041-98-5, 1-Naphthylamine, 4-(2-naphthylazo)- 65152-07-0, Phenol, 2,4-diisobutyl- 91588-45-3, m-Cresol, nitroso- 102763-39-3, Phenol, nitroso- 720688-66-4, Nicotine, compound with abietic acid 720688-66-4, Abietic acid, compound with nicotine

(antifouling properties of)

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L1: Entry 2 of 3

File: JPAB

Aug 20, 1982

PUB-NO: JP357135360A  
DOCUMENT-IDENTIFIER: JP 57135360 A  
TITLE: FOUL BREATH DETECTING AGENT

PUBN-DATE: August 20, 1982

INVENTOR-INFORMATION:

NAME

COUNTRY

MANSEI, KENJI

HARA, KENJI

IIJIMA, EIJI

ASSIGNEE-INFORMATION:

NAME

COUNTRY

KAO CORP

APPL-NO: JP56021278

APPL-DATE: February 16, 1981

US-CL-CURRENT: 600/529; 600/530  
INT-CL (IPC): G01N 33/50; A61B 5/00

ABSTRACT:

PURPOSE: To detect simply and sharply by utilizing the fact that dense blue color of the following reagent is decolored quickly under the existence of a foul breath component, by using 4, 4'-bisdimethylaminodiphenyl carbitol, especially a foul breath detecting agent containing said carbitol and a specific surface active agent.

CONSTITUTION: 4, 4'-Bisdimethylaminodipheyl carbitol (BDC-OH) designated by the formula is dissolved in water or a mixed solvent of acetone, ethanol and water in 0.005~1% concentration and said solution is kept at 2~7pH by using a buffer. The sensitivity is improved moreover by adding an anionic and amphoteric surface active agent to said solution to be 0.001~10% concentration. 0.5ml saliva is mixed with 0.5ml reagent and said mixture is left as it is at a room temperature for 25min and hereafter, is centrifuged. Absorbance of supernatant liquid is measured by 610nm wavelength and measured value is compared with a working curve using a standard substance dithioslatol having an SH radical preliminarily and then, the SH radical concentration of decomposed product of protein closely related to a foul breath in the saliva is measured. Hereby, the foul breath is detected accurately without differences between individuals.

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## ⑫ 公開特許公報(A)

昭62-151757

⑤ Int. Cl.<sup>4</sup>G 01 N 33/497  
31/22

識別記号

GAB

庁内整理番号

8305-2G  
8506-2G

④ 公開 昭和62年(1987)7月6日

審査請求 未請求 発明の数 1 (全3頁)

⑬ 発明の名称 口臭検知剤

⑰ 特 願 昭60-295567

⑱ 出 願 昭60(1985)12月26日

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## 明 細 書

## 1. 発明の名称

口臭検知剤

## 2. 特許請求の範囲

(1) 4,4'-ビスジメチルアミノジフェニルカルビノールおよび感度向上剤として、スルホン酸塩化合物、スルファミド塩化合物またはスルフィミド塩化合物を含有することを特徴とする口臭検知剤。

(2) スルホン酸塩化合物、スルファミド塩化合物またはスルフィミド塩化合物を口臭検知剤全重量に対して0.001~40重量%含有する前記第(1)項の口臭検知剤。

(3) 溶液状である前記第(1)項の口臭検知剤。

## 3. 発明の詳細な説明

## 発明の分野

本発明は口臭検知剤に関し、さらに詳しくは、4,4'-ビスジメチルアミノジフェニルカルビノールを口臭検知試薬とする、感度を向上させた口臭検知剤に関する。

## 発明の背景

従来、口臭の評価は人間の嗅覚を用いる官能評価法により行なわれてきた。しかし、この官能評価法は嗅覚の疲労や個人による評価のばらつきがあるなどの欠点があり、客観的な判定法としては問題があった。

近年、分析機器の著しい進歩により、燐や硫黄を含む化合物を選択的かつ高感度に検出する蛍光光度検出器(FPD)が開発され、この検出器を用いたガスクロマトグラフィーによる口臭の臭気成分の分析が行なわれた。その結果、口臭の強さが、口腔内の揮発性硫化物(以下、VSCという)量、特に、メチルメルカプタン量と相関関係にあることが判明した。しかしながら、ガスクロマトグラフィー法は、高価で特殊な装置が必要であり、また、調整が困難であるなどの問題があり、口臭を簡便に検知するには不適当である。

そこで、客観的かつ簡便な口臭検知法を開発すべく、種々研究がなされている。その一つとして、エム・エス・ロールバックハ、アナリティカル・

バイオケミストリー(M. S. Rohrbach, Anal. Biochem.) 52, 127 (1973)により報告されたメルカプト基定量試薬である4,4'-ビスジメチルアミノジフェニルカルビノール(以下、BDC-OHという)を用いる方法が検討され、唾液試料中のVSC量に基づいて口臭の検知を簡便に行なうための溶液や試験紙型の口臭検知剤が提案されている(特開昭57-135360号)。

しかし、この口臭検知剤においては、特に、BDC-OHのメルカプト基検出感度を高めるために、ある種の界面活性剤を加えることが提案されているが、界面活性剤の中には溶媒に対する溶解度の低いものも多く、溶液にするのが困難であったり、調製した溶液が界面活性剤のために攪拌等の操作により発泡するという問題がある。

本発明者らは、BDC-OHの口臭検知への適用の検討を行なう間に、ある種の含硫化合物がBDC-OHのメルカプト基検出感度を向上させ、これを用いることにより、界面活性剤を用いた場合のような発泡等の問題のない新規な口臭検知剤

ム、N-アルキルスルファモイル安息香酸ナトリウムなどのスルファミド塩化合物および安息香酸スルフィミドナトリウム、ジベンゼンスルホンイミドナトリウムなどのスルフィミド塩が挙げられる。

前記感度向上剤は、口臭検知剤全重量に対して0.001~40重量%、好ましくは0.1~20重量%の範囲で配合される。

つぎに、BDC-OHに対する種々の添加剤の感度向上効果を第1表に示す。なお、添加剤の効果は以下の方法により算出した指数で表わす。

BDC-OH 1mgをアセトン10mlに溶解して調製した溶液0.5mlおよび0.1M酢酸緩衝液(pH 5.0)3.5mlを混合し、ついで、0~10μgのメルカプトエタノールを含有する水溶液1mlを加えて5mlとし、610nmにて吸光度を測定した。第1図に示すように、610nmにおける溶液中のメルカプトエタノール量と吸光度の間には良好な相関関係が得られる。図に示された直線の勾配(以下、変化率という)はメルカプトエタノ

ール1μg当たりのBDC-OHの吸光度変化を表わす。

#### 発明の開示

本発明は、BDC-OHおよび感度向上剤としてスルホン酸塩化合物、スルファミド塩化合物またはスルフィミド塩化合物を含有してなる口臭検知剤を提供するものである。本発明の口臭検知剤は、唾液試料中のVSC含量を良好な感度で検知でき、客観的に、しかも界面活性剤を用いた場合のような泡立ちなどの問題なしに、簡便に、かつ、高感度で口臭を評価することができる。

本発明に用いられる感度向上剤としては、アントラキノンジスルホン酸ナトリウム、ナフトールジスルホン酸ナトリウム、ナフトキノンスルホン酸ナトリウム、トルエンスルホン酸ナトリウム、ベンゼンスルホン酸ナトリウム、フェノールスルホン酸ナトリウム、イセチオン酸ナトリウムなどのスルホン酸塩化合物、ベンゼンスルホンアミドナトリウム、N-アルキルベンゼンスルホンアミドナトリウム、スルファモイル安息香酸ナトリウ

ム、N-アルキルスルファモイル安息香酸ナトリウムなどのスルファミド塩化合物および安息香酸スルフィミドナトリウム、ジベンゼンスルホンイミドナトリウムなどのスルフィミド塩が挙げられる。

つぎに、添加剤を加えて、前記と同様にして吸光度を測定し、変化率を求めた(第2図参照)。これらの結果から、以下の式により指数を算出した。

$$\text{指数} = \frac{\text{添加剤を加えた時の変化率}}{\text{添加剤を加えない時の変化率}} \times 100$$

該指数は、添加剤を加えない場合のBDC-OHのメルカプト基検出感度を100とした場合の相対感度を表わしている。

第1表

添 加 剤	濃 度 (重量%)	指数
アントラキノンジスルホン酸ナトリウム	1	205
ナフトールジスルホン酸ナトリウム	1	195
フェノールスルホン酸ナトリウム	10	264
ベンゼンスルホン酸ナトリウム	10	209
トルエンスルホン酸ナトリウム	10	248
安息香酸スルフィミドナトリウム	10	419

第1表から明らかなごとく、BDC-OHにスルホン酸塩化合物やスルフィミド塩化合物を添加した場合、BDC-OHのメルカプト基検出感度が2~4倍上昇することがわかる。

かくして、本発明の口臭検知剤はBDC-OHおよび感度向上剤を、常法に従って、酢酸緩衝液、クエン酸緩衝液、ホウ酸緩衝液などの溶媒に溶解して溶液型の製剤とすることができ、あるいはこれを濾紙等に吸着、含浸させて試験紙型の製剤とすることができる。

たグラフであり、縦軸は吸光度を、横軸はメルカプトエタノール量( $\mu\text{g}$ )を表わす。各グラフ中、  
—○—○—は添加剤を加えない場合を、—●—●—は添加剤として安息香酸スルフィミドナトリウムを加えた場合を示す。

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本発明の口臭検知剤を適当量の唾液試料と混合あるいは試料に含浸し、ついで、37℃で所定時間インキュベートし、その変色を肉眼ないしは吸光度等で評価し、その変色の程度により口臭の有無を検知する。

#### 実施例

次に本発明の実施例を挙げるが、本発明はこれに限定されるものではない。

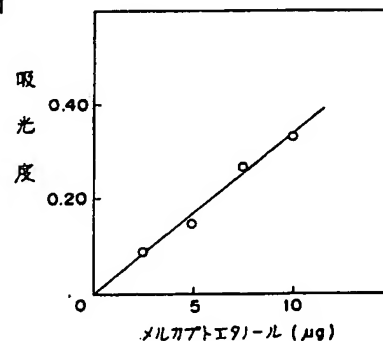
#### 実施例1

BDC-OH 1mgをアセトン10mlに溶解して調製した溶液0.5mlを安息香酸スルフィミドナトリウム500mg含有クエン酸緩衝液(pH5.0)4.5mlに加える。得られた口臭検知溶液および唾液をコンヴェー形拡散ユニットで37℃にて24時間インキュベートし、ついで該口臭検知溶液の吸光度を測定することにより口臭の評価を行なう。

#### 4. 図面の簡単な説明

第1図および第2図は、BDC-OHを用いてメルカプトエタノール量と吸光度の関係を測定し

第1図



第2図

